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Assessment of systematic errors in measurement of vapor pressures by thermogravimetric analysis

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Abstract

The present study explores the application of the diffusion limited evaporation theory to the estimation of vapor pressure from TG experimental data. A simplified method was developed to calculate the apparent values of the vapor pressure of pure substances from TG data, based on isothermal TG runs with crucibles having different surface areas available for evaporation. Antoine parameters are estimated through a numerical procedure based on a non-linear least square algorithm. The procedure also evaluates the substance diffusivity in nitrogen. The methodology developed might be used for a preliminary screening of the vapor pressure of pure compounds, due to the limited amounts of sample that are necessary and to the limited time frame required for the experimental runs. However, the estimation of diffusivity and vapor pressures values by the TG technique is possible with limited accuracy. Possible sources of error were thoroughly investigated and discussed.

Keywords: Thermogravimetric analysis; Evaporation rate; Vapor pressure; Diffusive evaporation; Data analysis

1. Introduction

Several techniques are currently used for measurement of the vapor pressure of a pure substance [1–3]. The vapor pressure is usually determined by either ebulliometric or static methods [1–6]. In ebulliometric (or dynamic) methods, the vapor pressure is determined by measuring the boiling temperature of the substance at various specified pressures or, as in comparative ebulliometry, comparing the vapor pressure to that of a reference substance [7–10]. In static methods, the vapor pressure established in a closed system at thermodynamic equilibrium is determined at a specified temperature [11–15]. These conventional techniques for vapor pressure measurement are highly accurate and reliable. However, they require rather large amounts of sample (millilitres) and are time-consuming. Several alternative experimental methods for vapor pressure determination are described in the literature, and include gas chromatography (GC) [16], effusion [17,18] and gas saturation methods [19–22]. Recently, a thermal analyzer was adopted as a transpiration apparatus for vapor pressure measurement [23].

Several attempts have been made to use thermal analysis techniques as alternative screening tools for the determination of vapor pressure [24–47]. The use of differential thermal analysis (DTA) and differential scanning calorimetry (DSC) was proposed [24–28], and a standard method for the measurement of vapor pressure by DTA or DSC was developed by the American Society for Testing and Materials [48]. Thermogravimetry (TG) was proposed as well for estimating the vapor pressures of pure substances [29–45] and of mixtures [46,47]. Compared with conventional techniques for vapor pressure measurement, thermogravimetry presents several advantages: the simplicity of the experimental set-up, the small amounts of sample (microlitres) and short experimental times required. However, the absence of sample mixing, the possible heat transfer and mass transfer limitations as well as the open configuration inherently limit the accuracy of the vapor pressure data that may be obtained by this technique. Moreover, the procedure for the estimation of vapor pressure data from experimental TG weight loss data is still controversial. Most of the former studies [31–41,44,45] proposed to derive information on vapor pressure from TG experimen-

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Nomenclature

A	Antoine parameter in Eq. (5)
A_c	surface available for mass transfer (m^2)
B	Antoine parameter in Eq. (5) (K)
C	Antoine parameter in Eq. (5) (K)
D	diffusivity (m^2 s^{-1})
k_0	diffusivity constant defined in Eq. (6) (m^2 s^{-1} K$^{-1.5}$)
L	crucible height (m)
N_b	overall molar bulk flow (mol s^{-1})
N_{ev}	evaporation rate (mol s^{-1})
P	total pressure (Pa)
P_{sat}	vapor pressure (Pa)
R	universal gas constant (J mol^{-1} K^{-1})
s	sample height (m)
t	time (s)
T	temperature (K)
y	molar fraction
y_s	molar fraction in the gas-phase at sample surface (see Fig. 4)
z	position on the z-axis in Fig. 4 (m)

Subscript

i	ith experimental run

tal data by the application of the Langmuir equation for free evaporation [49]. Pieterse and Focke [42] pointed out that the Langmuir equation neglects diffusive resistance to mass transfer, and proposed to base vapor pressure data calculation on the assumption of a diffusion limited vaporization process.

The present study explores the application of diffusion limited evaporation theory to the estimation of vapor pressure from TG experimental data. The diffusion limited evaporation assumption was used to develop a numerical method for TG data analysis, to yield correlation parameters for the vapor pressure as a function of temperature. The technique was based on the use of isothermal weight loss data obtained at different temperatures in different crucibles, providing different evaporating surfaces. The method was validated by comparing the experimental data obtained for several substances by the present approach to literature data obtained by conventional techniques. The validation evidenced the advantages as well as the limitations of the TG technique for the analysis of vapor pressure data.

2. Experimental section

2.1. Materials

The following sample materials were purchased from Aldrich (Milan, Italy): *N*-methyl-2-pirrolydone [CAS 872-50-4], 1,3-propanediol [CAS 504-63-2], dibenzyl ether [CAS 103-50-4], 2-naphthol [CAS 135-19-3] and salicylic acid [CAS 69-72-7]. Table 1 reports the physical and chemical properties of each substance.

2.2. Techniques

A TA Q-500 thermobalance was used to perform isothermal runs on each substance in a specific temperature range. Run duration was 30 min. A pure nitrogen purge flow of 100 ml/min (60 ml/min directly to the furnace and 40 ml/min to the balance and hence to the furnace) was used in experimental runs. The sample was positioned on the pan of the TG balance and then automatically inserted in the TG furnace. The temperature program typically consisted of a rapid heating (at a variable heating rate, lower than 50 K min^{-1}, selected by the control system of the TG device) followed by an equilibration at the desired temperature. The duration of this equilibration step depends on the final temperature, but is usually of less than 5 min.

Tests were performed using different types of cylindrical crucibles. Table 2 lists the relevant characteristics of the four crucibles used. Typical sample weights ranged between 9 and 36 mg, depending on the crucible surface and the density of the compound.

The temperature range was selected for each sample substance to obtain detectable weight loss rates. The choice of the upper limit of temperatures derived from the need to keep the test conditions well below the boiling point and to avoid decomposition phenomena. To verify the absence of relevant decomposition phenomena over the temperature range selected for isothermal runs, a TG–DSC–FTIR run (100% nitrogen, 60 ml/min purge gas flow, 10 °C/min heating rate) was performed on each sample substance using a Netzsch STA 409/C thermoanalyzer coupled to a Bruker Equinox 55 spectrometer [46,50].

3. Results and discussion

3.1. Experimental evaporation rates estimated in TG runs

Isothermal TG runs at different temperatures were carried out on the substances listed in Table 1 using all the crucibles

Table 1
Physical and chemical properties of the samples used in experimental runs

	N-Methyl-2-pyrrolidone	1,3-Propanediol	Dibenzyl ether	2-Naphthol	Salicylic acid
Formula	C_5H_9NO	$C_3H_8O_2$	$C_{14}H_{14}O$	$C_{10}H_8O$	$C_7H_6O_3$
Physical state	Liquid	Liquid	Liquid	Solid	Solid
Molecular weight	99	76	170	164	138
Density (kg/m^3)	1028	1053	1043	1280	1440
Melting point (°C)	−24	−27	4	122	159
Boiling point (°C)	202	215	296	285	280
Purity (%)	99.6	99.9	99.1	99.0	99.4

Table 2
Geometrical characteristics of crucibles used in isothermal TG runs

Type	Material	Internal diameter, ϕ_i (mm)	Open surface, A_c (mm^2)	Internal height, L (mm)	Ratio A_c/L (mm)
A	Al	6.0	28.3	4.7	6.0
B	Al	5.0	19.6	4.0	4.9
C	Stainless steel	5.2	21.2	5.0	4.3
D	Al	3.0	7.1	4.0	1.8

described in Table 2. Fig. 1 reports the results obtained for *N*-methyl-2-pirrolydone at 80 °C in crucible B. As shown in Fig. 1(a), at the beginning of the run, a limited temperature transient is present, during which the temperature of the sample reaches the set-point value. After the initial transient, the sample and furnace temperatures are maintained constant for the duration of the test. The weight of the sample steadily decreases during the run, as shown in Fig. 1(b), due to sample evaporation. The weight loss rate data, that represent the actual evaporation rate of the sample, reported in Fig. 1(b), show an initial increase during the start-up of the experimental run, due to the initial temperature increase to the set-point value. After the start-up, when temperature of the sample has reached the constant set-point value, the weight loss rate shows a decreasing value for the duration of the run. Qualitatively similar results were obtained in all the TG runs carried out on all the substances considered in the present study.

The results of isothermal TG runs performed in crucible B on *N*-methyl-2-pirrolydone at temperatures between 50 and 100 °C are shown in Fig. 2. The data reported in this and in the following figures were calculated as the mean of at least three experimental runs. As evident in the figure, the evaporation rate, and thus the weight loss rate given by the slope of the curves reported, increases with temperature. The weight loss rate at a given temperature is approximatively constant in the temperature range explored. A decrease in the slope of the weight loss curves and thus in the evaporation rate is actually present. Similar trends in weight loss as a function of temperature were found for all the substances in the present study.

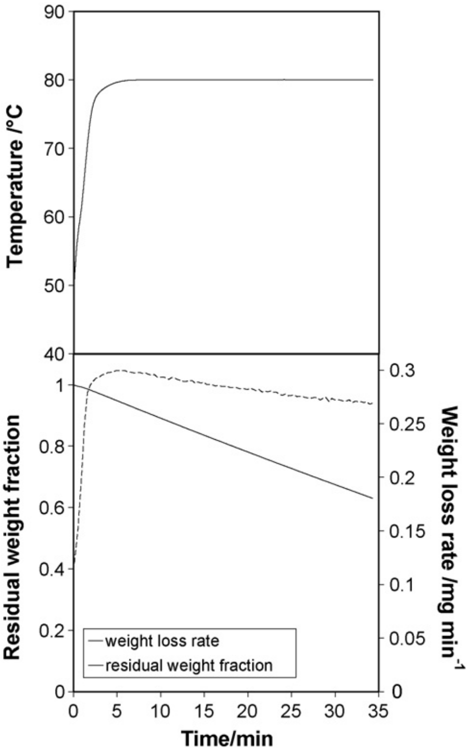

Fig. 1. Results of a TG isothermal run (80 °C, 100 ml/min pure nitrogen flow) on *N*-methyl-2-pyrrolidone in type B crucible.

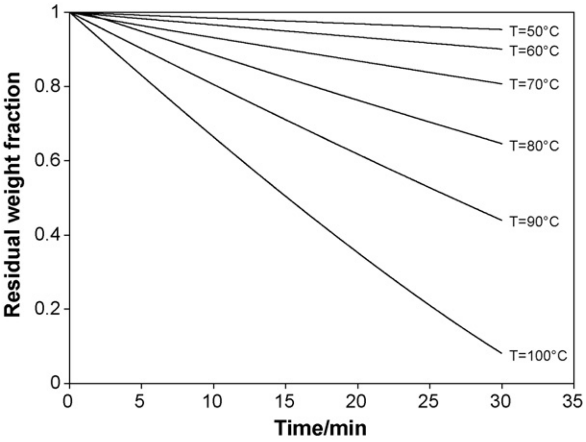

Fig. 2. Results of isothermal TG runs carried out on *N*-methyl-2-pyrrolidone samples at temperatures between 50 °C and 100 °C (100 ml/min pure nitrogen flow, type B crucible).

Fig. 3 reports weight loss rate data obtained in isothermal TG runs on 2-naphthol at 145 °C using different crucibles. The figure evidences that a higher ratio of crucible open surface, A_c, to the crucible height, L, results in a higher evaporation rate. Qual-

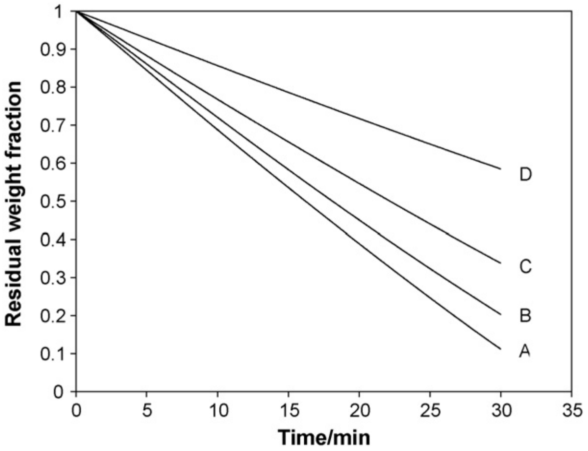

Fig. 3. Results of isothermal TG runs carried out on 2-naphthol samples at 145 °C in different crucibles.

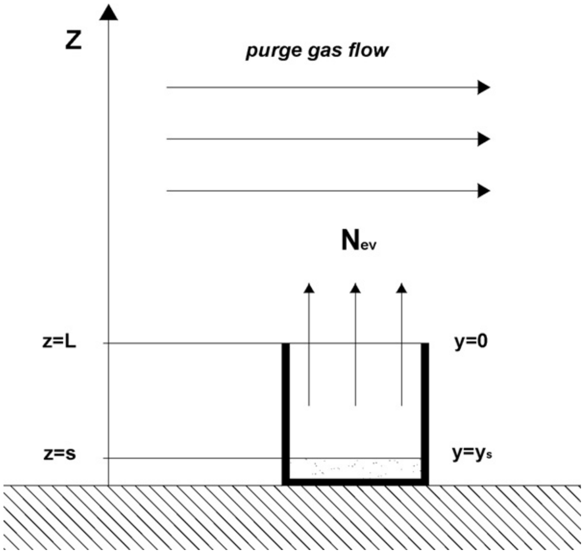

Fig. 4. Schematization of the mass transfer process during evaporation.

itatively similar results were obtained for the other substances investigated. The results confirm that the evaporation rate at a given temperature is influenced by the sample surface available for evaporation.

3.2. *Analysis of diffusive evaporation during isothermal TG runs*

Evaporation rates of low-volatility compounds in TG devices are controlled by diffusive phenomena through the gas film present between the liquid surface and the crucible edge [42]. The low purge gas velocities used in TG furnaces result in a limited influence of convective phenomena around the sample crucible. If the perturbations due to the purge gas flow around the crucible in the TG furnace are limited, a monodimensional analysis based on Fick's law may be applied [51] and the mass transfer process that takes place during the sample evaporation in TG isothermal runs may be schematized as shown in Fig. 4.

Fig. 4 reports an idealized representation of the evaporation process, used as the starting point for a simplified calculation of vapor pressure. Following the conventional approach to the modeling of diffusive evaporation processes, two assumptions were introduced in the analysis of the experimental data [51]:

(i) the system was considered perfectly isothermal;
(ii) a quasi-steady-state condition was assumed for the gas-phase concentrations and for the liquid level in the crucible.

The second assumption is valid for sufficiently low values of the vapor pressure [51], and is the more important, since it allows a steady-state analysis of the gas-phase diffusion process at a generic time t. Moreover, by this assumption the liquid level at time t may be considered constant and may be easily estimated by the sample weight provided by the TG balance, on the basis of the liquid density and of the crucible geometric data reported in Table 2. Experimental runs were carried out at atmospheric pressure, thus an ideal behaviour may be considered for the gas phase. The partial pressure of the evaporating compound may be calculated from the total pressure and the molar fraction in the gas phase. If the evaporation rate is sufficiently low, so that the liquid may be considered isothermal, the partial pressure of the evaporating compound at the liquid surface may be assumed equal to the vapor pressure of the substance at the test temperature. On the other hand, at the crucible edge, the partial pressure of the evaporating compound is approximately equal to zero if a sufficiently high purge flow is supplied.

On the basis of these assumptions, the differential molar balance on the z-axis (see Fig. 4) was derived from Fick's law [51]:

$$N_{\mathrm{ev}} = -A_{\mathrm{c}}\frac{DP}{RT}\frac{\mathrm{d}y}{\mathrm{d}z} + yN_{\mathrm{b}} \tag{1}$$

where N_{ev} is the molar flow of the compound due to the evaporation process, A_{c} the surface available for mass transfer, D the diffusivity of the compound in the gas mixture at the temperature of the experimental run, P the total pressure, y the molar fraction of the compound of interest, R the gas constant, T the temperature and N_{b} is the overall molar bulk flow. In the absence of thermal degradation, the overall bulk flow N_{b} equals the evaporative flow N_{ev}, and Eq. (1) may be rearranged as:

$$N_{\mathrm{ev}}dz = -A_{\mathrm{c}}\frac{DP}{RT}\frac{\mathrm{d}y}{(1-y)} \tag{2}$$

Integration of Eq. (2) between the evaporating surface of the liquid (see Fig. 4: $y = y_s$; $z = s$) and the crucible edge (see Fig. 4: $y = 0$; $z = L$) results in the following expression for N_{ev}:

$$N_{\mathrm{ev}} = -A_{\mathrm{c}}\frac{DP}{RT(L-s)}\ln(1-y_s) \tag{3}$$

where L is the height of the crucible, s the height of the liquid, and y_s the value of the molar fraction in the gas-phase at the liquid surface (see Fig. 4). The driving force in Eq. (3) may be directly expressed as a function of the vapor pressure P_{sat}:

$$N_{\mathrm{ev}} = -A_{\mathrm{c}}\frac{DP}{RT(L-s)}\ln\left(1-\frac{P_{\mathrm{sat}}}{P}\right) \tag{4}$$

Vapor pressures are related to the experimental TG data through Eq. (4). The overall molar evaporation rate may be directly calculated by dividing the experimental weight loss rate of the sample by the molecular weight of the evaporating substance. Temperature, pressure and crucible geometric data (A_{c}, L: see Table 2) have known and constant values. The liquid height, s, may be easily calculated from the sample weight and the liquid density.

Thus, Eq. (4) relates the experimental weight loss rate to the vapor pressure and the gas-phase diffusivity of the vapor in the purge gas. Unfortunately, values of vapor diffusivity in nitrogen as functions of temperature are usually unknown, although several models are available for their estimate (e.g. see [51,52] and references cited therein). On the other hand, the trends of both vapor pressure and gas diffusivity as a function of temperature are widely reported in literature. Vapor pressure at a given temperature, T, may be calculated from the Clapeyron equation

[5,53] or from the more widely used Antoine's empirical relation [5,52–54]:

$$\log_{10}(P_{\mathrm{sat}}) = A - \frac{B}{T + C} \tag{5}$$

where A, B, and C are the Antoine parameters, the temperature T is expressed in Kelvin and the vapor pressure P_{sat} is expressed in kPa.

Gas-phase diffusivity is known to vary with temperature following a power law, with an exponent between 1.5 and 1.75 [51,55]. In particular, if the Chapman–Enskog model [51] is considered, the following correlation may be used to approximate the variation of diffusivity with temperature:

$$D = k_0 T^{3/2} \tag{6}$$

where k_0 is a constant that may be estimated by the use of models for gas diffusivity [51,52], and is dependent on the properties of the substance and of the gas in which the diffusion takes place.

3.3. Estimation of vapor pressure from evaporation rate data

The above approach may be used for calculation of vapor pressure even in the absence of data on vapor diffusivity. The experimental TG data on the evaporation rate, N_{ev}, obtained at different temperatures and with different crucible types, are the starting point to calculate the Antoine parameters and the k_0 value. The correlation between the measured evaporation rate, the geometric constraints and the Antoine parameters to be used in Eq. (5) may be evidenced substituting Eqs. (5) and (6) in Eq. (4):

$$N_{\mathrm{ev}} = -A_{\mathrm{c}} k_0 \frac{PT^{1/2}}{R(L-s)} \ln\left[1 - \frac{1}{P} 10^{(A-B/(T+C))}\right] \tag{7}$$

A non-linear least-squares best-fit procedure may be used to estimate the Antoine parameters and the diffusivity constant. The following objective function may be defined to apply the least-squares procedure:

$$\begin{aligned} F &= \sum_{i=1}^{n} \left[N_{\mathrm{ev},i}^{\mathrm{exp}} - N_{\mathrm{ev},i}^{\mathrm{mod}}\right]^2 \\ &= \sum_{i=1}^{n} \left[N_{\mathrm{ev},i} + A_{\mathrm{c},i} k_0 \frac{PT_i^{1/2}}{R(L_i - s_i)} \ln\left[1 - \frac{1}{P} 10^{(A-B/(T_i+C))}\right]\right]^2 \end{aligned} \tag{8}$$

where n is the total number of experimental runs available, and the subscript i ($i = 1,2,\ldots,n$) identifies the values of N_{ev}, T, A_{c}, L and s in the ith experimental run. Substitution of the diffusivity with the approximate expression in Eq. (6) in the objective function leads to the inclusion in the constant k_0 of any deviation of the overall mass transfer coefficient from that present in the ideal laminar flow conditions to which Fick's law applies.

The relevant number of unknown parameters (4) that need to be estimated from experimental runs requires a sufficiently extended experimental data set. In particular, experimental data over a significant range of temperatures and evaporating surfaces must be available to obtain reliable estimates of the parameters. However, the maximum temperature used in the experimental runs should be sufficiently below the boiling temperature of the substance, so that the above assumptions are valid.

To validate this procedure, the experimental data collected in the present study were used to estimate the vapor pressure, the Antoine parameters and the diffusivity of the substances listed in Table 1. Calculated values of the vapor pressure were compared to the available literature data. A comparison between the apparent diffusivity calculated by Eq. (6) and the values obtained from available literature models for the estimation of gas diffusivity was also performed.

The Antoine parameters reported in the literature and the value of the k_0 constant in Eq. (6) calculated by literature models are compared in Table 3 to values obtained from the analysis of experimental data. The table also reports the temperature ranges selected for the experimental runs. The best-fit Antoine parameters calculated from TG experimental data are quite different from those retrieved from the literature. Fig. 5 compares the vapor pressure values calculated with literature coefficients and

Table 3
Experimental intervals seleted for TG runs and comparison of Antoine parameters calculated from TG data with those reported in the literature

		Temperature interval (K)	A	B (K)	C (K)	k_0 ($\mathrm{m^2/s\,K^{1.5}}$)	$k_{0,\mathrm{exp}}/k_{0,\mathrm{mod}}$	Mean temperature of the experimental runs (K)
N-Methyl-2-Pyrrolidone	Literature	361–477	7.4038	2570.3	0	1.86×10^{-9}	–	–
	Experimental	323–373	4.92	946.3	−153.8	2.29×10^{-9}	1.231	348
1,3-Propanediol	Literature	332–488	8.3476	3149.9	9.1444	2.08×10^{-9}	–	–
	Experimental	353–383	9.63	2994.9	−56.8	2.24×10^{-9}	1.077	368
Dibenzyl ether	Literature	413–561	6.8432	2507.3	−43.15	1.18×10^{-9}	–	–
	Experimental	393–428	4.01	750.5	−222.2	1.84×10^{-9}	1.559	410.5
2-Naphthol	Literature	401–561	7.2293	2827.5	−19.868	1.52×10^{-9}	–	–
	Experimental	398–438	3.05	467.5	−257.6	2.02×10^{-9}	1.329	418
Salicylic acid	Literature	446–504	5.5381	1050.0	−228.14	1.66×10^{-9}	–	–
	Experimental	433–458	1.43	35.6	−397.8	4.64×10^{-9}	2.795	445.5

Table 4
Relative and absolute errors calculated in the estimation of vapor pressure from TG data

	Temperature range (K)	Maximum relative error (%)	Mean relative error (%)	Maximum absolute error (kPa)	Mean absolute error (kPa)
N-Methyl-2-pyrrolidone	323–373	25.03	13.06	0.70	0.30
1,3-Propanediol	353–383	28.30	16.30	0.50	0.20
Dibenzyl ether	393–428	12.10	6.20	0.20	0.09
2-Naphthol	398–438	3.70	2.10	0.07	0.03
Salicylic acid	433–458	9.23	3.11	0.50	0.20

those calculated by TG data. As shown in the figure, higher deviations were obtained for all the substances at higher values of the vapor pressure. This general trend should be expected, since, as discussed above, at higher temperatures more relevant deviations are expected from ideal laminar flow and diffusive evaporation. These deviations are likely to result in higher apparent values of the diffusivity and of the vapor pressure calculated by the present approach.

Table 4 shows that for all the substances considered the mean relative error present among the literature values of the vapor pressure and those calculated from the TG data obtained in the present approach is always below 20% and usually of about 5% for most of the substances considered in the present study. The maximum relative errors are always below 30%. The corresponding mean and maximum absolute errors in vapor pressure estimate are always below 0.3 and 0.7 kPa, respectively. Thus, as expected, the accuracy of vapor pressure estimation by TG data resulted far lower than that of the conventional techniques used for vapor pressure measurement.

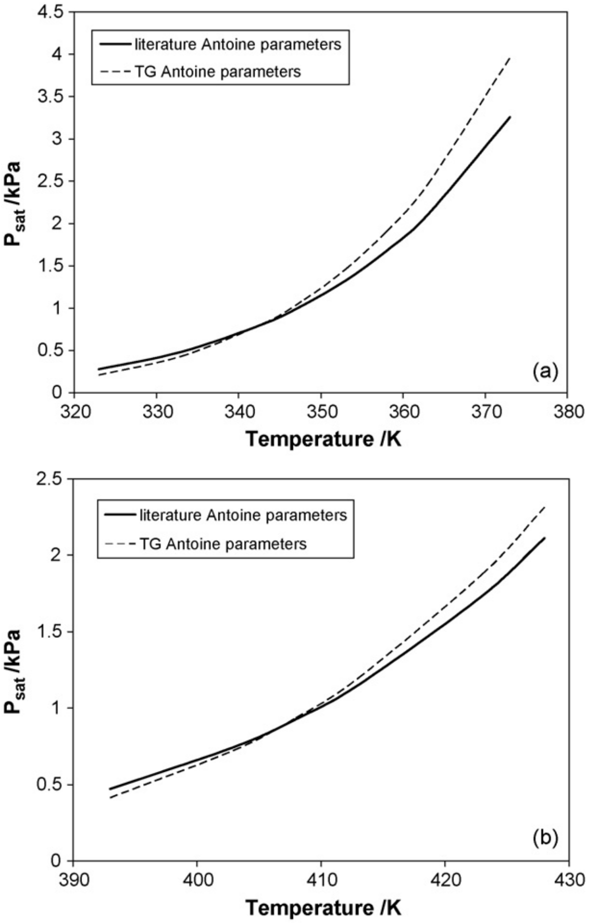

Fig. 5. Comparison of vapor pressure values estimated from experimental TG data and using literature values for the Antoine parameters. (a) *N*-Methyl-2-pyrrolidone, (b) dibenzyl ether.

Comparison of the experimental values estimated for the vapor diffusivity to the model values shows that the approach is able to estimate at least the order of magnitude of the diffusivity. As shown in Table 3, the differences between the experimental best-fit and model values of k_0 are lower than a factor 3. The model values of k_0 were calculated at the mean temperature of the experimental runs for each substance considered, that was also reported in the table.

It is important to understand the origin of the deviations between the experimental and model data. As expected, the experimental apparent diffusivity, that was estimated by Eq. (7), resulted higher than that obtained by the literature model. As remarked above, the form of Eq. (7) leads to include in the diffusivity constant k_0 any deviation of the overall mass transfer coefficient from that of ideal laminar flow conditions. Thus, the non-ideal conditions present in the TG furnace lead to slightly higher mass transfer coefficients, that result in the estimate of higher apparent values of vapor diffusivity. This explanation is confirmed by Table 3, that reports the ratio of experimental to model k_0 values as a function of the mean temperature of the experimental runs performed for each substance. As shown in the table, the deviation of the experimental apparent values of diffusivity with respect to model values increases with temperature (see the mean temperature of experimental runs, last column of the Table). Since the experimental runs were performed with a constant purge gas flow, the purge gas velocity inside the TG furnace increases with temperature, due to the decrease in gas density. As expected, the higher deviations in the apparent diffusivity are present at higher temperatures, where the higher gas velocities cause higher deviations from the ideal laminar flow schematized in Fig. 4 and assumed as a starting point for the analysis.

4. Conclusions

A simplified method based on diffusive evaporation modeling was developed to estimate the apparent values of the vapor pressure of pure substances from TG data. Even if the results confirmed that TG analysis may by no way substitute conventional methods for vapor pressure measurement, the methodology developed seems suitable for a preliminary screening of the vapor pressure of pure compounds, and may be attractive in the presence of concurrent evaporation and decomposition phenomena [56]. The experimental validation of the procedure showed that the direct estimation of diffusivity and vapor pressure values by the TG technique is possible, with mean relative errors usually far below 15%, while the maximum relative error is in general below 25%. Thus, non negligible inaccuracies may affect the results of direct vapor pressure measurements by TG analysis. However, the results obtained evidence that informative and significant data may be obtained if the conditions of experimental runs are carefully controlled (low purge gas flow, low temperatures) and if a sufficiently extended data set is used for the calculation of Antoine parameters. Since the sources of error seem to be mostly due to the deviations of the fluidodynamic regime around the crucible from diffusion controlled evaporation, the development of empirical calibrations based on adimensional parameters may be a suitable route to improve the quality of vapor pressure data obtained by TG analysis.

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